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14.3: Sintering Ceramic Laser Materials with a High Power 83 GHz Beam*

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Millimeter-wave processing (sintering) of ceramic laser host materials has been under investigation at the Naval Research Laboratory (NRL) for high energy laser (HEL) applications. Advantages of polycrystalline, compared to single-crystal laser host materials, include lower processing temperature, higher gain from higher dopant concentrations, cheaper fabrication, and larger devices. Millimeter-wave processing has been shown to be an effective alternative to conventional vacuum furnaces for pressure-free sintering of low-loss oxide ceramic materials. It involves direct volumetric heating of the workpiece. This often results in superior microstructure with fewer trapped pores, cleaner grain boundaries, and smaller grain size than conventionally sintered materials. These properties are critical to achieving high optical quality laser host materials. The absorbed power per unit volume in a ceramic is proportional to the microwave frequency ω according to

$$P_{\text{absorbed}}(\omega, T) = \frac{1}{2} \omega \epsilon_0 \epsilon''(\omega, T) |E|^2 \text{ where } \epsilon_0 \text{ is the free}$$

space permittivity, ϵ'' is the relative dielectric loss and E is the local rf field. Thus the power loss is a function of both the temperature T of the ceramic and the frequency; at a given frequency, oxide ceramics tend to be more absorbing at higher temperatures, and at a given temperature, an oxide ceramic is more absorbing at higher frequencies.

Equipment. The NRL gyrotron-based material processing facility (*cf.* Fig. 1) features a 15 kW, CW, 83 GHz GYCOM gyrotron oscillator, which can generate between 1 W/cm² and 2 kW/cm² irradiance. The facility features a quasi-optical beam system for beam focusing and manipulation and an inner vacuum chamber for atmosphere control and vacuum processing. The gyrotron is operated via a fully automated computerized control system written in the LabVIEW™ platform with feedback from

extensive *in-situ* instrumentation and visual process monitoring. The system has the capability of sample rotation and translation during processing.

Experimental setup and processing. The green compacts are uniaxially pressed to densities ranging between 50 and 60% of theoretical density (TD) and may be cold isostatically pressed (CIPed) as well, as high green density is a prerequisite for effective sintering. The ceramic work pieces are placed in an open or closed crucible and are directly exposed to the 83 GHz beam which is focused to a roughly elliptical shape (approximately 1 cm by 4 cm) by the concave mirror. This type of beam is adequate for processing the small compacts currently being tested (diameter \sim 0.5 cm) and allows two samples to be processed together under similar conditions. Larger compacts will require a larger, more uniform beam. The crucible and the materials surrounding the workpiece (casketing) are chosen to provide thermal isolation and low temperature heating, and to reduce radiative losses to the cold-walled furnace. Analytic and numerical models are used to guide the design of workpiece casketing and will be discussed. The ceramic work piece may be embedded in a setter powder and/or microwave susceptor. Zirconia is often used as a setter powder and other setter powders include boron nitride, alumina, and yttria. The sample is placed inside a small boron nitride crucible both to protect it from contamination and to provide a more uniform thermal bath. The beam power and intensity at sintering temperatures is a few kilowatts and a few 100 W/cm², respectively. The mirror position is adjusted during processing to optimize irradiation of the workpiece. The workpieces are processed in a small vacuum chamber (inner diameter 33 cm, height 28 cm) (*cf.* Fig. 2) in a vacuum of between 25 and 100 milli Torr. The pressure is monitored for signs of outgassing during initial heating. A resistive heater

is available for pre-sintering heat treatments. The temperature is monitored by both an S-type thermocouple (platinum/platinum with 10% rhodium) situated near the sample and a remotely located two-color pyrometer. These temperature monitors can easily give quite different results unless care is taken to avoid large temperature gradients. YAG and Ytria, potential ceramic laser host materials, have been sintered with millimeter-

wave beams with up to 99% theoretical density. So far translucent samples have been achieved, but not with the transparency needed for lasing. Several factors impact the quality of the sintered material including the presence of agglomerates, impurities, processing atmosphere, sintering aids, and thermal gradients. Efforts to improve the transparency will be discussed.



Figure 1. NRL 83 GHz gyrotron-based material processing facility. The millimeter-wave beam furnace is located inside the large processing chamber which serves to confine the millimeter-wave radiation. A 2-color pyrometer looks down on the furnace through a screen at the top of the processing chamber. The millimeter-wave beam exits the gyrotron horizontally and is deflected by a slightly concave mirror into the vacuum furnace through a quartz window.

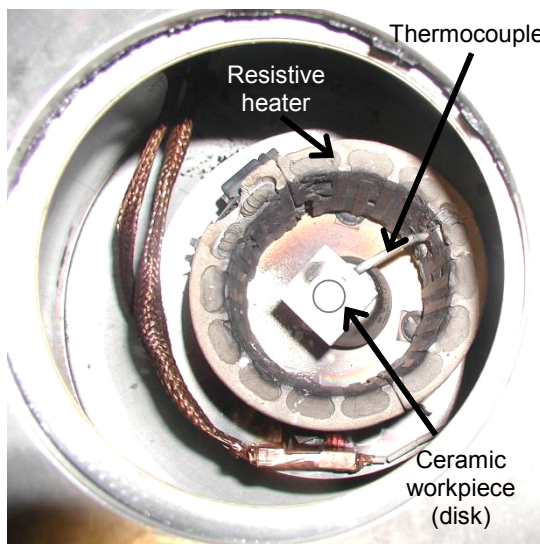


Figure 2. Millimeter-wave beam furnace. Side view [left], top view with lid including quartz vacuum window removed [right].

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